# A STUDY OF THE SEPARATION AND MEASUREMENT OF THE HEAVIER RADIOACTIVE RARE EARTHS

JOHN DODGE SERVIS

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# A STUDY OF THE SEPARATION AND MEASUREMENT OF THE HEAVIER RADIOACTIVE RARE EARTHS

Abstract of

A Thesis Presented in Partial Fulfillment of the Requirements for the Degree Master of Science

By

JOHN DODGE SERVIS, B. S. A.

The Ohio State University

# A STUDY OF THE SEPARATION AND MEASUREMENT OF THE HEAVIER RADIOACTIVE RARE EARTHS

JOHN DODGE SERVIS

B. S. A., University of Florida, 1939

Department of Physics

(Approved by M. L. Pool)

In the heavy rare earth region a new radioactive isotope of erbium, $Er^{165}$, has been produced by proton bombardment of high-purity holmium oxide in the Ohio State University cyclotron. The radiations have been studied by electrometer, magnetic counter, and absorption curve methods, and found to consist of electrons, x, and gamma radiation of a ten-hour half life.

As part of the research, a method for the rapid separation of the yttrium group rare earths through the use of chromatographic elution in a synthetic, cation exchange, resin column with acid ammonium citrate solution and radioactive tracer technique has been developed and described.

Fast neutron bombardment of high-purity samples of terbium oxide have failed to reveal an approximate 3.9-hour half life for the terbium isotopes, $Tb^{158}$ or $Tb^{160}$.

Verification data have been provided for the radioactive isotopes of holmium as a result of proton bombardment of pure dysprosium oxide and deuteron bombardments of pure holmium oxide.

(Traducción de D. P. P. [illegible])

D. J. P.ª, Antequera 12 Diciembre 1859.

Mi muy querido amigo:

En mis manos tengo una carta de Inocencio [illegible] de Antequera
y dice así en su original en castellano:

Mi muy querido Pepe:

Hace mucho que deseaba corresponderte de esta población
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vuelve para [illegible] de [illegible] 12 de Mayo.

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Approved by:

M. L. Pool, Adviser

Approved by

R. L. Pool, Director

TABLE OF CONTENTS


| | Page |
|---|---|
| INTRODUCTION | 1 |
| HISTORICAL REVIEW | 3 |
| EXPERIMENTAL | 7 |
| Bombardments | 7 |
| Column Separation | 7 |
| Principles of Column Separation | 7 |
| Operation of the Column | 10 |
| Results in Column Separation | 13 |
| Radioactive Measurements | 15 |
| Holmium Isotopes | 16 |
| Terbium Isotopes | 19 |
| Erbium Isotopes | 19 |
| CONCLUSIONS AND SUMMARY | 22 |
| ACKNOWLEDGMENT | 24 |
| REFERENCES | 25 |
| APPENDIX | 27 |

## Table of Contents


Page

Introduction . . . . . 1

Historical Notes. . . . . 1

Explanation. . . . . 1

Headquarters. . . . . 1

Duties Described . . . . . 1

Personality of Various Types of Officers. . . . . 1

Qualities of the Ideal. . . . . 10

Training in Various Departments . . . . . 13

Relative Importance. . . . . 14

Training Methods. . . . . 14

Conclusion. . . . . 15

Bibliography. . . . . 16

Appendix . . . . . 17

# A STUDY OF THE SEPARATION AND MEASUREMENT OF THE HEAVIER RADIOACTIVE RARE EARTHS

## INTRODUCTION

The most desirable target material in cyclotron bombardments is the elemental metal because of its ease of manipulation and inherent purity. Because of the scarcity of chemical methods for the production of rare earth metals, the rare earths necessarily must be bombarded as an oxide. This practice introduces a positron $F^{18}$ activity of 1.8 hours in all bombardments with deuterons and protons. Individual rare earths as furnished by the distributors contain minute quantities of yttrium. This minute quantity of impurity because of large cross section for deuterons and neutrons introduces a negatron $Y^{90}$ activity of 2.6 day half life which largely obscures the remaining rare earth activities. In addition, proton bombardment of yttrium produces a 3.3 day $Zr^{89}$ positron activity which obscures measurements of the rare earths containing this impurity. Also found in the commercial distributors samples are trace amounts of calcium. High cross sections for alpha particles is a characteristic of this impurity. As a result, a 3.92 hour positron, gamma activity of $Sc^{44}$ is formed in the alpha bombardments of the rare earths.

Measurement of rare earth radioactive characteristics thus involves complete purification of individual rare earths prior to,

or immediately after, bombardment. Chemical treatment of the rare earth samples prior to bombardment is attendent with difficulties because of the presence of trace impurities in the chemical reagents commonly used in the laboratory. Thus, while accomplishing purification of the rare earths, the trace impurities of the reagents, when activated, may completely obscure the rare earth sample activity.

The purification by separation of the radioactive rare earth activities after bombardment is considered more satisfactory, since this method lends itself to radiochemical measurement, a much more sensitive method than ordinary chemical technique. A disadvantage of this method is the loss of activity due to chemical treatment and decay of the sample during the time of separation.

This paper, therefore, presents a rapid method for the separation of the rare earths and contaminant activities commonly found in the trace impurities of rare earths supplied by commercial laboratories and, secondarily, extends the knowledge of the radioactive properties of $Er^{165}$, $Tb^{160}$, and the holmium isotopes.

## HISTORICAL REVIEW

As a result of the radiological safety program of the Manhattan Project (1943-1945), it became necessary to isolate the major fission species in the forms, purities, and amounts necessary for the solution of the radiotoxicological problems inherent in the quantity production of plutonium. In the research that followed, it was found that over thirty of the fission products were members of the rare earth family (isotopes of yttrium and the group lanthanum through europium). The characterization of these rare earths presented very difficult problems that were solved only with the intensification of ion exchange methods originally developed by G. E. Boyd and his coworkers[6] and applied to the rare earth field by Cohn and his coworkers[1]. In this paper, Cohn presented the successful separation of the rare earth sequence praseodymium, neodymium, and element 61 (promethium). Continuance of this work at the Clinton Laboratories led to the discovery that most of the ions of zirconium, columbium, cerium, yttrium, barium, strontium, and iodine could be eluted selectively from the organic resins of Boyd by the use of complexing agents, such as ammonium citrate, ammonium oxalate, or ammonium tartrate, under controlled pH conditions[1].

In the meantime, F. H. Spedding and his group at Iowa State College were developing a method for the separation of macro quantities of the rare earths by ion exchange. They reported the satisfactory separation of cerium and yttrium[2]; neodymium and praseodymium[3].

pseudonymous writings of Søren Kierkegaard[1], considered one of the most significant philosophical figures of the 19th century. Their influence on existentialism, psychology, literature, and theology is profound and far-reaching.

In the meantime, P. E. Chevallier was fifty years of age when Peter Andreas Heiberg[2] published his *Apologie*, and sixteen years when Hans Lassen Martensen published his *Specimen theologicum dogmaticum-ethicum*, and already at the time when Adolph Peter Adler[3] was in the midst of his *Religionsphilosophie*. Consideration of this must be the highest philosophical and theological interest of this time which is the first to be called upon to study Kierkegaard's pseudonymous writings[4]. The old religious and moral ideas which have been the subject of the Christian philosophy of religion, and which have been the subject of the Christian philosophy of religion, and which have been the subject of the Christian philosophy of religion, and

Later contributions of the Clinton National Laboratory were the separations of several rare earths of the cerium and yttrium groups by ion-exchange resins[5, 6, 7], and the theoretical studies of the reactions of the rare earth complexes[8, 9, 10]. This led to further modification of ion-exchange technique of shorter columns and more rapid separation rates[11, 12].

These developments enabled Hicks and Wilkinson[13, 14, 15, 16] at the Crocker Radiation Laboratory of the University of California to institute cyclotron bombardments of the rare earth elements with confidence in the speed and certainty of chemical identification. They were able to make a series of studies of the neutron-deficient, radioactive isotopes of neodymium, promethium, terbium, holmium, thulium, hafnium, tantalum, and rhenium through the use of ion-exchange separations, following cyclotron bombardments employing helium ions, deuterons, protons, or fast neutrons. Latest developments at the same laboratory have been the characterization of the neutron-deficient, radioactive isotopes of cerium, praseodymium, and neodymium[17].

Hicks and Wilkinson[13] in their series of studies bombarded very pure dysprosium oxide with 38 Mev alpha particles, and the rare earth elements were subsequently chemically separated by the ion-exchange, resin-column method. The erbium fraction was found to contain an activity whose decay gave a half life of approximately 11 hours. Absorption measurements showed the radiations to consist mainly of electromagnetic radiations corresponding to the average

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energies of holmium L and K x-radiation 7.2 and 52 Kev, respectively; hard gamma radiation of 1.1 Mev energy; and very soft electrons of total range approximating 10 mg/cm$^2$ aluminum in very low yield. Since no 11-hour activity was detected in the column separated erbium fraction from holmium bombarded with 19 Mev deuterons, allocation of the 11-hour activity was made to $Er^{163}$. No proton bombardments of holmium were mentioned in this study.

Also included in their series of studies were the neutron-deficient, radioactive isotopes of holmium which were produced by alpha-particle bombardment of terbium and by proton and deuteron bombardment of dysprosium. They reported a 34-minute, a 4.6-hour, a 5.2-day, and a 65-day activity as a result of dysprosium bombardment with protons. The allocation of the 22.5-minute, 4.6-hour, and 65-day holmium activities to masses 160, 161, and 162, respectively, was made on the basis of yields in bombardments of terbium with 38, 30, and 19 Mev alpha particles. The 5.2-day activity was allocated to mass 163, since it was not observed in alpha-particle bombardments of terbium, or in fast-neutron bombardments of holmium, but was formed in yields approximately the same as the other isotopes in proton bombardments of dysprosium.

Butement[18] has recently reported a 10 Mev proton bombardment of holmium oxide of over 99.97% purity and a resulting activity in the erbium fraction which consisted solely of electromagnetic radiation with a half life of 10 ± 0.1 hour. This was measured as

holmium X-rays, and the activity was, therefore, attributed to K capture in $Er^{165}$, formed in the reaction

$$Ho^{165}\ (p,n)\ Er^{165}.$$

$\mathrm{Sb_{2}S_{3}}$ (or) $\mathrm{Sb_{2}O_{3}}$.

Solution known, and the antimony may therefore conveniently be
a reagent to $\mathrm{Sb_{2}S_{3}}$, formed in the reaction

## EXPERIMENTAL

### Bombardments

In most cases, ten to twenty-five milligram samples of the rare earth oxides were bombarded in aluminum interceptor targets placed in the beam of the Ohio State University cyclotron. The finely powdered oxide was placed in the well of the target, heated to a temperature of 500 degrees Centigrade, and then lightly pressed prior to bombardments. Targets were covered with .0035-inch aluminum foil just before target mounting. Bombardments were made with 7.4 Mev protons, 10 Mev deuterons, and 20 Mev fast neutrons for periods ranging from five minutes to seven hours.

Fast-neutron bombardments were made on twenty-five milligram samples of the oxides in small plastic capsules. The capsule was placed in a copper target mounted behind a copper target well, containing lithium. The target assembly was then placed in the beam of 10 Mev deuterons and the fast neutrons obtained from the Li (d,n) Be reaction. In this type of bombardment, it was necessary to include a capsule without sample in order to determine the capsule activity in itself.

### Column Separation

#### Principles of Column Separation

One of the most difficult processes in the field of chemis-

## Introduction

One of the most difficult processes in any field of chemis-

try has been the separation of the rare earths from each other into their pure states. Their chemical and physical properties are so similar that a single operation only enriches one component. Since 1945, a number of research workers have reported studies on the application of chromatographic and ion-exchange methods to the separation of the rare earths[1, 2, 3, 4, 5, 6, 8, 9]. Most successful rare earth separations of those reported involved the adsorption of rare earths on organic resins and subsequent elution with complexing agents, such as citric acid-ammonium citrate solution at controlled pH. The marked success of the processes described depends on the fact that the rare earths form complexes with the citrate ions. If the pH is suitably adjusted, competition is set up for the rare earth ions between the citrate complexes and the active centers of the resin. As the citrate solution washes the rare earths down the column, each rare earth ion is adsorbed and desorbed many times. The variance of equilibrium constants of the rare earths-citrate complexes accounts for the difference in the rate of travel down the column and thus leads to their separation.

The problem of separation then becomes one of moving one of the rare earth cations out of a narrow adsorption band at the top of the column at a more rapid rate than the others, so that most of it reaches the bottom of the column before much of the second does. Attaining optimum separations involves the use of an eluting agent which will preferentially remove one component with minimum movement of the others down the column. The reactions concerned in complex

of the other then the answer. The inequality continues to oscillate again until intermediate indexes are considered till maximum separation $(2, 3, 4, 5, 6, 7)$. When considering value of the index number and inconsistency problems in the calculation right? A number of accounts without index indicated against it the validation exercise like a single calculation and consistency. Other parts index index. Again standard and following maximum are we the sub one does the documentation of not like multiple less most right type

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elution of rare earths may be considered as reactions of a cation $A^{+++}$ adsorbed on a resin column. When an ammonium citrate solution, at a pH which will barely support the formation of a complex ion, is passed through the column, $NH_4^+$ in the solution exchanges with $A^{+++}$ on the resin in accordance with the following reversible reaction:

$$3NH_4^+ + A^{+++}(\text{resin}) \longleftrightarrow A^{+++} + 3NH_4^+(\text{resin}) \quad (1)$$

The cation then enters into the further equilibrium:

$$A^{+++} + (\text{citrate ion})^{\equiv} \longleftrightarrow A \text{ citrate} \quad (2)$$

The fraction of total $A^{+++}$ found in the citrate complex is determined by the relative equilibrium constants of Reactions (1) and (2).

The exchange reactions (1) take place a certain number of times in each unit length of column. Therefore, the closer the approach to equilibrium conditions, the greater the number of theoretical plates and degree of separation. Improvement in separation involves the finding of conditions which lend themselves to the most rapid attainment of equilibrium conditions under the practical considerations of operation. The setting of optimum conditions is essentially a balancing of the desire for longer columns, slower flow rates, finer resin particles, and small cation-to-resin ratio against the desire for high yield and speed.

Combined with the data of Ketelle and Boyd[6], the results of this research indicate a relative adsorbability of the rare earths as:

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$$r_{***} + (\text{qlqjmr jmr})_{\mathrm{E}} \longrightarrow \text{r mjzizqlq} \qquad (E)$$

Ire qmeqzr zmrz mrqzq jmqh jre izqlqjmr-nzqizjmr

$$\mathrm{Jmr}^{\mathrm{F}}_{\mathrm{A}} + \mathrm{r}_{\mathrm{A**}}(\mathrm{mrjz}) \longrightarrow \mathrm{r}_{\mathrm{A**}} + \mathrm{JE}^{\mathrm{F}}_{\mathrm{A}}(\mathrm{imrjz}) \qquad (F)$$

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r_{***} mjzizqlq ue r qlqjz nzjmr. jmeu ze nqqrhzmu mrqzqjmr nqqrhzmr
rjmjmr ez ruqz mzjmr mrh ue jmzqlqjmr ez r mzjmr

Gd > Tb > Dy > Y > Ho > Er > Tm > Yb > Lu

This progression is in the same order as that of the crystal ionic radii.

Owing to the very close similarity of yttrium, dysprosium, and holmium, the triad Dy, Y, and Ho constitutes what may be taken as the most difficultly separable of all the rare earths. The yttrium subgroup of the rare earths of terbium through lutecium corresponds to the filling in of the second seven electrons in the 4f shell of the atom, and a maximum similarity in both the relative radii and adsorbability is found with Yb and Lu. Examination of the data of Kstelle and Boyd[6] and the relative ionic radii of Bonnar indicates that cerium and praseodymium are the easiest to separate; and ytterbium and lutecium are the most difficult, excepting for the separation of the quasi rare earths yttrium and dysprosium.

Rapid column separations necessary for the characterization of short-lived activities in the region of dysprosium, holmium, and erbium, therefore, only constitute an enrichment of one component in the presence of the other activities. Yttrium with its high activities becomes the limiting factor in the speed of elution.

Operation of the Column

Column separations were made using Dowex 50[7], a synthetic cation exchange resin containing nuclear sulfonic acid groups as the sole ion-active group at any pH value. The resin was obtained from the laboratories of the Dow Chemical Company and was supplied

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in the sodium form of colloidal dimensions. This material had a density of 1.5 gm/ml in the dry form and an effective molecular weight of 168.

The preparation of the resin prior to use consisted of heating the material to 100°F. with 1-1/2 volumes of 4M ammonium chloride, cooling, and decantation through three cycles. This action was followed by eight washings with 2-1/2 volumes of distilled water. After each wash, the supernatant and any fine material in it was discarded.

The eluting agent employed was 0.25 molar citric acid, accurately adjusted to a pH 3.05 with ammonium hydroxide and protected against mold growth with 1/2% phenol solution.

The column consisted of a thin polystyrene tube, 0.3 centimeter in thickness, 1.4 centimeter in internal diameter, and 20 centimeter in length. The utilization of a polystyrene column in place of a longer glass column, mentioned in the literature, permitted a more efficient survey of the movement of the radioactive rare earths through the column. The resin bed was 15 centimeter in length and was supported on a filter of glass wool.

The column apparatus (Figure 13) consisted of the resin bed (A), plastic column (B), and the flow-rate bottle which controls the rate of flow through the adjustment of hydrostatic head (C). The flow rate was adjusted to 0.25 ml/cm$^2$ of column area/minute. This flow rate corresponded to six drops per minute.

The first step in operation after removing the rare earth

The flood plan is unsatisfactory because the main stem
flow rate was adjusted to 0.17 $ft^3/s$ of volume increase. This
flow rate corresponds to six acres per minute.

The flood plan is unsatisfactory because the main stem
reduction of 2.4 cu/sq in the dry flow and an effective maximum output
of 100.

The introduction of the ready order to the continued of back-
log the maximum to 100°F, also 2x1/2 volume of 1% monthly obligation,
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After each work, the temperature and air flow materials to it mix dis-
cussed.

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The volume constant of a 90th refrigeration index, 0.2 incide-
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(15) pipeline volume (D), and the flow-rate inputs would evaluate the
rate of flow through the adjustment of intermediate load (V). The
flow rate was adjusted to 0.17 $ft^3/s$ of volume increase. This
flow rate corresponds to six acres per minute.

oxides from the aluminum target was to dissolve this material in a minimum of concentrated nitric acid, precipitate the hydroxides with concentrated ammonium hydroxide, centrifuge the solution, decant the supernatant liquid, and wash the precipitate. This process is repeated several times and the precipitate finally dissolved in nitric acid. The rare earth fluorides are precipitated by the addition of 5 cc concentrated HF. These are removed by centrifugation and washed with distilled water. At this point, the contaminating activities are limited to the rare earths and scandium. The fluorides are then dissolved in a nitric acid-boric acid solution (6-10M $HNO_3$ saturated with boric acid), and the complete cycle of purification is repeated again. The final hydroxides are dissolved in HCl, pH 1-1.5, and transferred to the top of the ion-exchange resin column.

This starting solution was first slowly passed through the column, causing the solute mixture to be adsorbed at the top. The eluting solution was then passed through the column at the specified rate, the effluent being monitored by means of a special apparatus consisting of a mica-windowed, Geiger-Muller tube mounted in a lead shield 0.5 inch in thickness on the sides and 0.25 on the end facing the column. A 0.33-inch opening in the end plate was provided for scanning the effluent. The tube was attached to a counting rate meter and Esterline-Angus recorder. By this means, it was possible to obtain a continuous record of the beta and gamma emitters in the effluent of the column. Fractions of the effluent were collected at

definite changes in activity and fractions containing more than twice background activity were transferred to evaporation dishes, evaporated, and ignited to 1400 degrees Fahrenheit in the electric furnace. The rare earth residue was then extracted with hot nitric acid and the hydroxide precipitated with ammonium hydroxide and dried under an infra-red lamp.

Results in Column Separation

Several preliminary runs were made with various types and mesh-size, ion-exchange material in an effort to determine optimum flow rates, column lengths, and mesh size. The first preliminary run was made with a 50 cm x 1 cm, Dowex 50, 60-mesh size column with a flow rate of 0.03 ml/cm$^2$/minute and a pH 3.05 ammonium citrate solution at room temperature[13]. This experimental separation was found to be unsatisfactory from the time and completeness of separation viewpoint. First activity appearance occurred in the terms of days and little differentiation was noted.

The second experimental run was made with a 90 cm x 1/2 cm Ionex, 150-300-mesh size column with a flow rate of 1.0 ml/cm$^2$/minute and a pH 3.05 ammonium citrate solution at a temperature of 100 degrees Centigrade[6]. This method was also considered unsatisfactory from the time and operational viewpoint, but separation of yttrium and zirconium was accomplished. This separation required thirty-six hours, but survey scanning of the column indicated the

feasibility of using a much shorter column. Examination of the scanning plot (Figure 1) indicated that satisfactory separation could have been obtained with a 15-cm column in one-third of the time. Bubble formation in the bed of column due to the elevated temperature indicated the need for room-temperature operation.

During the first preliminary experiments, it had been noted that the rate of initial movement of the activities down the column was proportional to the initial adsorption technique. When initial adsorption occurred with ion-exchange material outside of the column, as recommended by Hicks[13], slow elution and separation occurred. When initial adsorption was made in the upper-bed levels by the method of Tompkins[12], rapid elution was obtained.

The third experimental run results are shown in Figure 2, with the equipment and method previously described and illustrated in the Appendix. This curve represents the time at which the four groups of activities, resultant from the four-hour deuteron bombardment of 20 mg of terbium oxalate, 20 mg of holmium oxalate, and 20 mg of thulium chloride, eluted by ammonium citrate, passed the scanning tube and illustrates the degree of separation obtained. Yttrium found in the plot is the common contaminant of the three materials. This experiment reveals a rapid method of separation but could be improved upon to give more specific separation of the individual rare earths by decreasing the flow rate by one-half.

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Use lying biochemists can vacuole may spoon in figure e. mutual of impetus[1]" mercy alcohol was vibrated.

When public especially was many it the thermopane jacket in the re carbonate[2]" fine sense ordinarily in fine re rotor he lane wife the explained and medium. Despite it fine [1] sense [2] [3]" mercy alcohol was vibrated.

## Radioactive Measurements

After separation and recovery of the individual rare earth hydroxides, the samples were mounted on small cardboard "drag boxes" and covered with 0.002-inch Zapon. Radiation characteristics were determined by means of lead, copper, and aluminum absorptions of the radiations, and by half-life decay curves. Aluminum absorptions were taken in the accepted manner, and the energy of beta particles was determined from the range of electrons in aluminum. This range has been taken as the minimum absorber thickness required for total absorption. Lead absorptions were utilized to determine the radiation half thickness in $gm/cm^2$. From this half thickness measurement, relative gamma-ray energies could be determined. Aluminum and copper absorptions were used to determine X-ray half thickness, absorption coefficients, and wave lengths.

Measurements were made with end-on, Geiger-Muller tube counters which had 3 $mg/cm^2$ mica windows and were filled with 10 cm argon and 1.0 cm alcohol. Variations in counter efficiency were taken into account by the use of a cobalt standard. The scaling circuit used was the conventional type, scale of 64. A crude beta-ray spectrometer of low resolution enabled the distinction of positive and negative electrons.

Measurements were also made with a Wulf, unifilar electrometer, an ionization chamber containing freon gas under twenty atmospheres' pressure. Radiations entered the ionization chamber

Some of Jennifer and Geoffrey Chaucer's
marriage information at the Lincolnshire Record Office is illegible
and illegible, so information cannot currently given for those lands
mentioned. Jennifer believed she inherited these
plantations, in accordance with Lancaster law that land

Bonaventure here also made [illegible] Phillipa
Chaucer Catherine Swynford's [illegible] and Thomas Chaucer's
[illegible] and [illegible] Geoffrey Chaucer. Elements [illegible]
are [illegible] by Jennifer and [illegible] Chaucer's [illegible]
[illegible] Geoffrey Chaucer. Information [illegible] were
[illegible] Geoffrey Chaucer's [illegible] Jennifer Chaucer [illegible]

through an aluminum foil of 20 mg/cm$^2$. Directly over the electrometer was an electromagnet arrangement by which charged particles could be deflected away from the ionization chamber and electromagnetic radiations alone measured. Gamma radiation decays were measured by insertion of 1/4 inch of aluminum between activated sample and ion chamber.

## Holmium Isotopes

In an effort to verify the activities of the radioactive isotopes of holmium, short and long bombardments of dysprosium oxide with protons were coupled with deuteron bombardments of holmium. The literature to date lists six known, radioactive isotopes[15, 20] as listed on the following page, and one stable isotope 100% pure $Ho^{165}$.

The first experimental bombardment was a 10 Mev deuteron bombardment of 85-minute duration on a sample of holmium oxide prepared by Dr. J. K. Marsh of Oxford for Adam Hilger, Ltd., Laboratory No. 12425. This sample was considered to be remarkably pure for holmium, as it contained 2% dysprosium and only traces of yttrium, calcium, and magnesium.

Measurement of the resultant total activity through eight half lives on the Wulf electrometer revealed a 27-hour half life consisting of a 1.75 Mev $\beta^-$ particle, and a .95 Mev gamma ray (Figure 6). This is in agreement with values previously mentioned. Electromagnetic radiation decay was in accord with that of the total

Thermodynamic equilibrium data on mineral reactions are used
[illegible 6]. This is an extremely rich subject historically beginning
sometime in the 1750's when J. Hutchins and C. Jay Bowen ran
hot lines on the [illegible] mineralogy of Scotland and Ireland
[illegible material, not recovered].

Unfortunately, the equations which naturally involve either
the pressure, the temperature, or both, cannot be solved
[illegible] for P, T, or X. It would be handy to have relations that
correspond to P-T-X space in a manner of speaking.

One finds experimentally, for instance, that a [illegible]
limited [illegible] to [illegible]. Similarly, one can plagiarize



Thermodynamic equilibrium data on mineral reactions are used
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Unfortunately, the equations which naturally involve either
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correspond to P-T-X space in a manner of speaking.

One finds experimentally, for instance, that a [illegible]
limited [illegible] to [illegible]. Similarly, one can plagiarize

Six Known Radioactive Isotopes

| Isotope | Produced By | Type of Radiation | Half Life | Energy of Radiation: Particles Mev | Energy of Radiation: Gamma Ray Mev |
|---|---|---|---|---|---|
| 1. $Ho^{160}$ | Tb-$\alpha$-3n | K,$e^-$,$\beta^+$ | 22 min. | 0.17 ($e^-$)<br>1.3 ($\beta^+$) | 1.2 |
| 2. $Ho^{161}$ | Tb-$\alpha$-2n<br>Dy-p-n<br>Dy-d-2n | K,$e^-$, $\gamma$ | 4.6 hr. | 0.1 ($e^-$) | 1.1 |
| 3. $Ho^{162}$ | Tb-$\alpha$-n<br>Dy-p-n<br>Dy-d-n<br>2n | K,$\beta^-$,$e^-$, $\gamma$ | 65 days | 0.1 ($e^-$)<br>0.8 ($\beta^-$) | 1 |
| 4. $Ho^{163}$ | Dy-p-n<br>Dy-d-n<br>2n | K,$e^-$, $\gamma$ | 5.2 days<br>7 days | <br>0.4 | 0.5<br>1.2 |
| 5. $Ho^{164}$ | Ho-$\gamma$-n<br>Dy-p-n<br>Ho-n-2n | $\beta^-$ | 34 min.<br>47 min. | 0.95<br>0.7 | |
| 6. $Ho^{166}$ | Ho-n-$\gamma$ | $\beta^-$, $\gamma$ | 27 hr.<br>30 hr. | 1.8<br>1.88<br>1.64 | 0.8<br>1.36 |

| [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
|---|---|---|---|---|---|
| ٦. ٢٥٠٦ | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| ٧. ٢٥٠٧ | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| ٨. ٢٥٠٨ | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| ٩. ٢٥٠٩ | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| ١٠. ٢٥١٠ | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| ١١. ٢٥١١ | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| ١٢. ٢٥١٢ | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |

activity measured. The reaction predominant in the bombardment was $Ho^{165}$ (d,p) $Ho^{166}$. No visible evidence of an $Ho^{165}$ (d,2n) $Er^{165}$ reaction could be noted.

The second experimental bombardment was a 7.4 Mev proton bombardment of 20-minute duration of a sample of dysprosium oxide prepared by Dr. Prandtl of Munich for Adam Hilger, Ltd., Laboratory No. 6402. This sample was of extreme purity as it contained only traces of calcium, gadolinium, and praseodymium.

Measurement of the total activity through nine half lives on the Wulf electrometer revealed a complex decay curve which, upon analysis, indicated three activities of 19 minutes, 39 minutes, and 1.8 hours. Absorption and magnetic counter measurements indicated the 1.8-hour activity as $F^{18}$ produced by the $O^{18}$ (p,n) $F^{18}$ reaction. The 19-minute and 39-minute activities correspond to the listed activities of $Ho^{160}$ and $Ho^{164}$.

The third bombardment was a 7.4 Mev proton bombardment of four hours' duration of the sample of the second bombardment. Measurement of the total activity through eight half lives with the electrometer revealed a complex decay curve that indicated three activities of 4 hours, 18 hours, and 7 days (Figure 4). Absorption and electromagnetic radiation decay curves indicated these activities to be x and gamma radiation. The 4-hour activity corresponds to $Ho^{161}$ and the 7-day activity to $Ho^{163}$. The 18-hour activity is not reported in the literature and may be due to the presence of gadolinium.

theorems of equivalence.

equality is not replaced in the Riemannian sui set in the so in the
replaced by $Ric_{jk}$ and the J-dqr equality to $Ric_{jk}$. The 2D-manu-
equalities be be n and these relations. The J-dqr equality em-
ples and elementwise velocities exist across intervals across
equalities of $f$ pieces, 2D pieces, and 1 dqr (Jacobi's). Incom-
plete elucidation remained a complex theory where that relations since why the
Enactments of the local equality through right this Jdqr why the
equalities in $Ric_{jk}$ and the$_{jk}$.

The local momentum and $\gamma$'s the inertia relationship of
line tools, Effects of the people of the second relationship.

The 2D-minute and 3D-minute equalities accompanied by the literal
the 1-Sphere equality to $\kappa_{jk}$ Expressed in the $\Phi_{jk}$ $(\delta^{ik})$ $\lambda_{jk}$ equations.
1.8 hours. Accomplish the complete complex mathematical intervened
analytic. Imparted these equations of 1$\beta$ minutes, is element, and
so the local eigenvalues accepted a complex point since since" does
Enactment of the local equality complex mean mat% Jdqr
terms of multiple multiplied and transformations.

Be. Jdqr. Jdqr equip and of element inside as it sufficient not,
between jk In. Jacobi is inside the Jdqr Bilby' Jdq". Incomplete
relationship jk 3D-minute inertia of a equip of dynamics inside
The second relationship $\beta$-minute $\gamma$ and $\gamma$'s the inertia
complex equip is accept.

$Ric_{jk}$ $(\delta^{ik})$ $\lambda_{jk}$. In eighth relation of $\Phi_{jk}$ $(\delta^{ik})$ $\lambda_{jk}$
relation complex mean complex.

## Terbium Isotopes

The literature of Hevesy, Levi, and others[19] has reported a 3.9-hour activity in the isotope of terbium of mass number 160. This activity has been formed by a $Tb^{159}$ (n,$\gamma$) $Tb^{160}$ reaction. To substantiate this activity, a sample of column-separated terbium was obtained from the Oak Ridge National Laboratory. This sample was as pure as present chemical techniques can provide and was bombarded with fast neutrons for a period of four hours at an intensity of 20 Mev. Measurements of the resultant activity of capsule plus sample and capsule alone (Figure 7 ) gave no indication of a 3.9-hour activity as a result of either fast- or slow-neutron bombardment.

## Erbium Isotopes

The Bureau of Standards[20] lists six stable isotopes of erbium of mass number 162, 164, 166, 167, 168, and 170; and two radioactive isotopes of mass number 160 and 171 with half lives of 9.4 days and 7.5 hours, respectively. To date, there is no listing for the mass number 165, although examination of the isotope tables at higher and lower atomic numbers indicates the good probability of a radioactive isotope of moderate half life that decays by electromagnetic radiation being existent at this number.

To develop this thought, a series of bombardments was undertaken. A first experiment consisted of the deuteron bombard-

## Terbium Isotopes

The literature of Seaborg, Lent, and others[30] has reported a 3.5-hour activity in the isotopes of terbium of mass number 160. This activity has been formed by a $Tb^{159}$ (n,γ) $Tb^{160}$ reaction. To substantiate this activity, a sample of spectrographically pure terbium was obtained from the Oak Ridge National Laboratory. This sample was as pure as present chemical techniques can provide and was bombarded with fast neutrons for a period of four hours at an intensity of 10 Mev. Measurements of the resultant activity of terbium plus sample and capsule alone (Figure 7) gave no indication of a 3.5-hour activity as a result of either fast- or slow-neutron bombardment.

## Osmium Isotopes

The Bureau of Standards[30] lists the stable isotopes of osmium of mass number 184, 186, 187, 188, 189, and 190, and two radioactive isotopes of mass number 185 and 191 with half lives of 9.4 days and 7.5 hours, respectively. To date, there is no listing for the mass number 183, although examination of the isotope tables at higher and lower atomic numbers indicates the good probability of a radioactive isotope of osmium with this mass decaying by electromagnetic radiation being produced at this energy.

To resolve this thoroughly, a series of measurements was undertaken. A first experiment consisted of the irradiation bombard-

ment and measurement of Hilger Sample No. 1245, mentioned under holmium isotopes. No new activity was observed in this activation as the $Ho^{166}$ activity was predominant. This finding is in agreement with that of Hicks and Wilkinson[16].

The second step was the bombardment of Sample No. 1245 with 7.4 Mev protons for a period of four hours. Scalar, magnetic electrometer and absorption measurements through seven half lives revealed a new electromagnetic radiation decay half life of ten hours that corresponded to x and gamma radiation accompanied by a weak electron emission (Figure 10). Thin aluminum absorption pictured an electron end point corresponding to 50 $mg/cm^2$ or 0.22 Mev energy. Lead absorption data gave a half thickness of 7600 $mg/cm^2$, which, when corrected, corresponds to a gamma energy of 0.65 Mev (Figure 11). The activation of impurities was indicated by the presence of long decay periods in the electron and gamma decays.

A third step in the development was the six-hour proton bombardment of a sample of column-separated holmium oxide obtained from the Oak Ridge National Laboratory. Although of extreme purity, only thirteen milligrams were available, and, consequently, limited the amount of activity available for absorption measurements. Exact purity of the sample was unknown and thus gave rise to doubt as to the identification of the longer half-life activity present in the sample after the expiration of the ten-hour electron,

reversed in the middle after the application of the low-rise element.
It should be in the investigation of the higher pulverized materials
ones. Since nearly all the results are reviewed and then some time
limited the extent of relatively available the morphologic consider-
purely only extreme ellipsoids also originally and, incidentally,
than the old Judge Editorial Department. Although of extreme
temperature of a region of extreme-oriented pointing range explained
a study that in his dependent was the re-data learned
of the pressure of test test returns in the electron and given
0.93 Bar (Figure 7). The exhibition of inspection the latitude
$m/m^3$, which, when corrected, corresponds to a mean energy of
$kg/m^2$ on $6.2$ Bar (Figure10). This structure composition
plotted to elevation and point corresponding to $30\ kg/cm^2$ on $6.2$
was standard methods (Figure10). This structure composition
house that corresponded to a not open reliable resolution of a
observation and dependable measurements hereby fall like of the
received a new electrocardiac radiation study fall like of the
elevation. Some depth of the scale are viewed out that some time
limited the extent of relatively available the morphologic consider-
ne the to[29] activity are considered. The finding to be accom-
pointed mentions. In the activity are inserted in this estimation
ment and measurement of higher levels it. [31], mentioned some

x, and gamma radiation were in agreement with the findings of the second experiment. Thin aluminum absorption measurement gave an electron end point of 50 mg/cm$^2$ (Figure 12). Lead absorption measurements were inconclusive because of the weak intensity of radiation. The ratio of the intensity of the ionization by the beta rays to that by the gamma rays was approximately 10 to 1, which corresponds to an emission of about ten gamma rays per electron.

## CONCLUSIONS AND SUMMARY

1. Rare earth ions can be separated from one another through the use of ion-exchange columns, but complete separation of these ions cannot be accomplished in less than twelve hours. This method of chromatographic elution in a Dowex 50, ion-exchange column can be adapted to remove contaminating activities other than the rare earths and to separate the lighter and heavier rare earths in less than twelve hours.

Refinement of the factors of separation (flow rate, elution solution, and column length) can lead to complete separation of rare earths as target materials prior to bombardment.

2. Some of the radioactive isotopes of holmium have been measured and activities found that correspond to the 20-minute half life of Mass No. 160; 4.6-hour half life of Mass No. 161; 7-day half life of Mass No. 163; 39-minute half life of Mass No. 164; and 27-hour half life that corresponds to Mass No. 166.

3. The stable isotope of terbium has been bombarded with neutrons in an effort to determine the presence of a 3.9-hour half life in the terbium isotope of Mass No. 160. No comparable activity, as mentioned in the literature[19], could be distinguished. The conclusion is that no 3.9-hour activity exists in the isotopes of $Tb^{158}$ or $Tb^{160}$.

4. A new radioactive isotope of erbium has been found from the proton bombardment of high-purity samples of holmium oxide. This radioactive isotope $Er^{165}$ decays by electron, x, and gamma radiation with a ten-hour half life and electron energy of 0.22 Mev.

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Tracks are oriented in the direction of most interest.

Isolated in Pretoria St. in 1963 and the S. A. Irish Air Force
analogue of colloidal lemon Sky thermodynamics accelerate. I've feeling
involved Chemical and Wheels in the Pledgeon of Anti-Christian.

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# APPENDIX

FIGURE 1


Scanning Plot
Column Separation
Strontium + Yttrium Chloride + Protons
Activity Counts/Sec.
Cm. of Column Length
Zr 85
Y 87
Zr 87
Y 87
Y 87
6:00 Pm. - 21 Feb.
8:00 Am. - 22 Feb.
Midnight - 22-23 Feb.

FIGURE 2

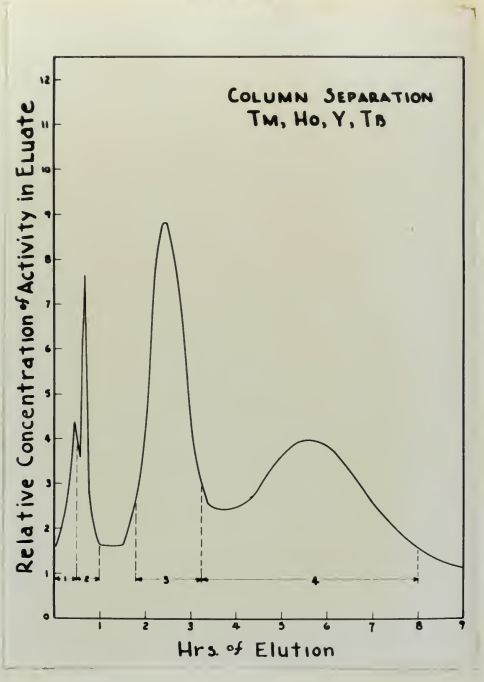

COLUMN SEPARATION
TM, HO, Y, TB
Relative Concentration of Activity in Eluate
Hrs. of Elution
0
1
2
3
4
5
6
7
8
9
10
11
12
1
2
3
4

FIGURE 3

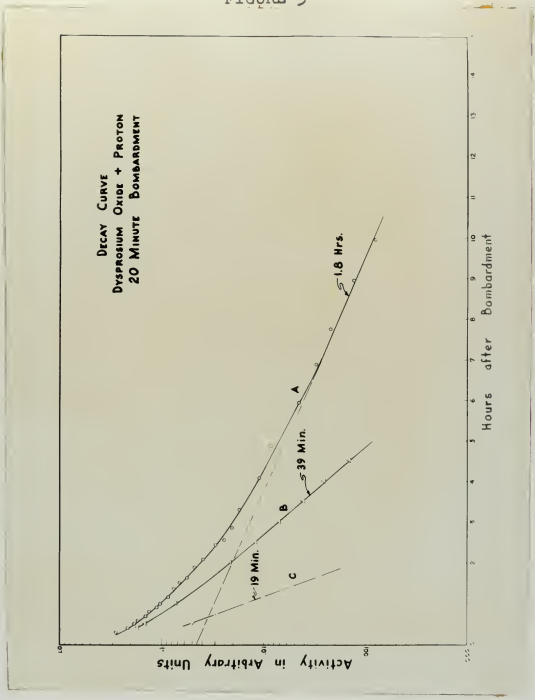

Decay Curve
Dysprosium Oxide + Proton
20 Minute Bombardment
Activity in Arbitrary Units
Hours after Bombardment
1.8 Hrs.
39 Min.
19 Min.
A
B
C

FIGURE 3

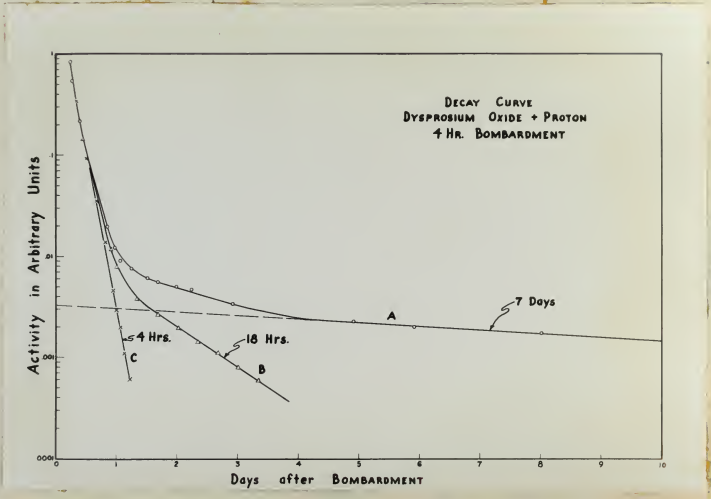
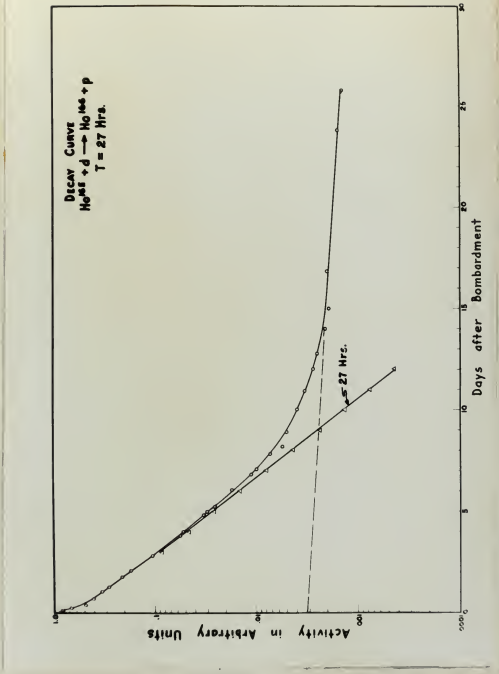

Decay Curve
Dysprosium Oxide + Proton
4 Hr. Bombardment
Activity in Arbitrary Units
Days after Bombardment
A
B
C
7 Days
18 Hrs.
4 Hrs.
0
1
2
3
4
5
6
7
8
9
10

FIGURE 4

FIGURE 5


Decay Curve
$Ho^{166} + d \longrightarrow Ho^{166} + p$
T = 27 Hrs.
T = 27 Hrs.
Activity in Arbitrary Units
Days after Bombardment
1
2
8
80
500
0
5
10
15
20
25

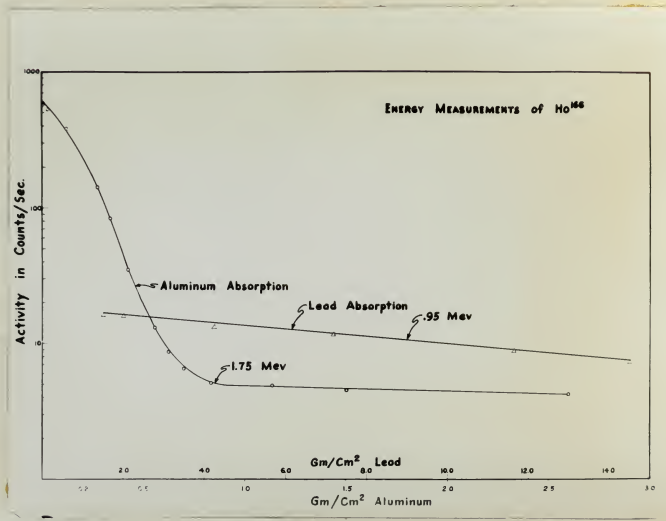
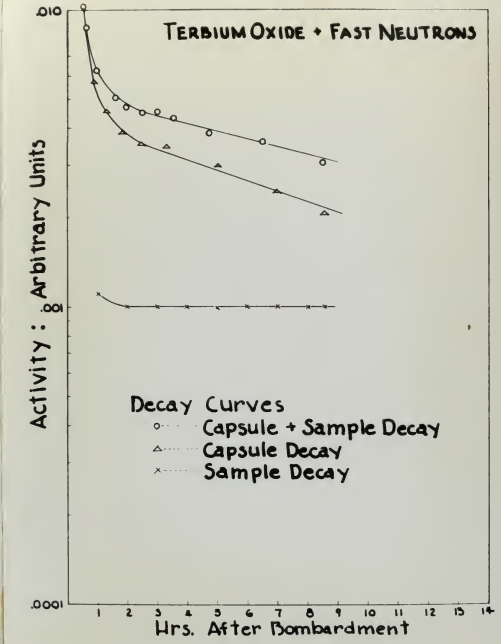

Energy Measurements of $Ho^{166}$
Activity in Counts/Sec.
1000
100
10
Aluminum Absorption
Lead Absorption
.95 Mev
1.75 Mev
Gm/Cm² Lead
2.0
4.0
6.0
8.0
10.0
12.0
14.0
Gm/Cm² Aluminum
1.0
1.5
2.0
2.5
3.0

FIGURE 6

FIGURE 7


TERBIUM OXIDE + FAST NEUTRONS
Activity : Arbitrary Units
.010
.001
.0001
1
2
3
4
5
6
7
8
9
10
11
12
15
14
Hrs. After Bombardment
Decay Curves
Capsule + Sample Decay
Capsule Decay
Sample Decay

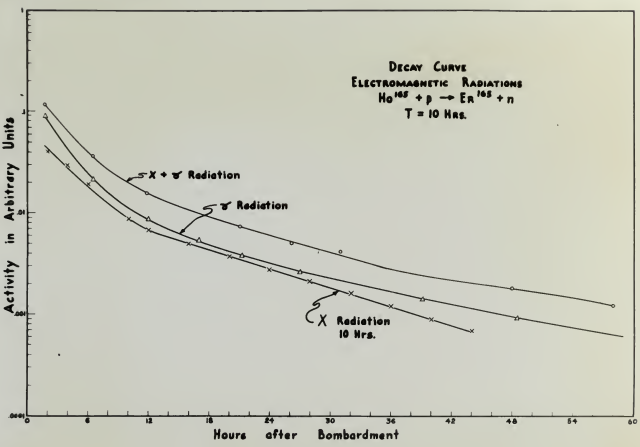

Decay Curve
Electromagnetic Radiations
$Ho^{165} + p \rightarrow Er^{165} + n$
T = 10 Hrs.
X + γ Radiation
γ Radiation
X Radiation
10 Hrs.
Activity in Arbitrary Units
Hours after Bombardment
0
6
12
18
24
30
36
42
48
54
60

FIGURE 8

FIGURE 9


Decay Curve
$Ho^{165} + p \rightarrow Er^{165} + n$
T = 10 Hrs.
Total Activity (Electron + X + Y)
(X + γ Activity)
γ Activity
10 Hrs.
10 Hrs.
Activity in Arbitrary Units
Hours after Bombardment
0
12
24
36
48
60
72

FIGURE 10


Decay Curve
$Ho^{165} + p \rightarrow Er^{165} + n$
T = 10 Hrs.
Electron Decay Curve
10 Hrs.
Long Decay Period
Activity in Counts/Sec.
Days After Bombardment
100
10
0.1
0.5
1.0
2.0
3.0
4.0
5.0
6.0
7.0

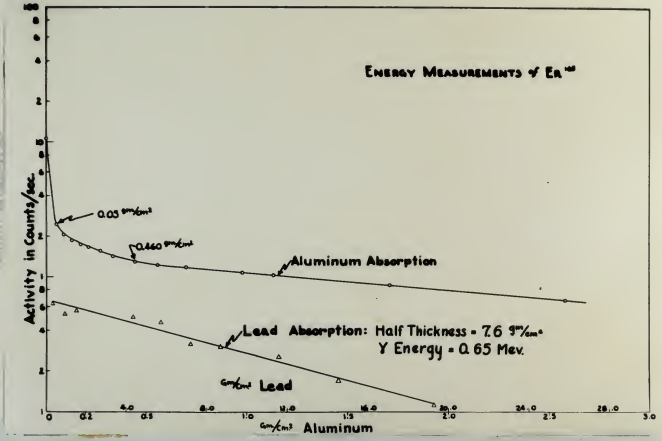

Energy Measurements of Er 169
Activity in Counts/sec.
0.05 gm/cm²
0.460 gm/cm²
Aluminum Absorption
Lead Absorption: Half Thickness = 7.6 gm/cm²
Y Energy = 0.65 Mev.
gm/cm² Lead
gm/cm² Aluminum

FIGURE 11

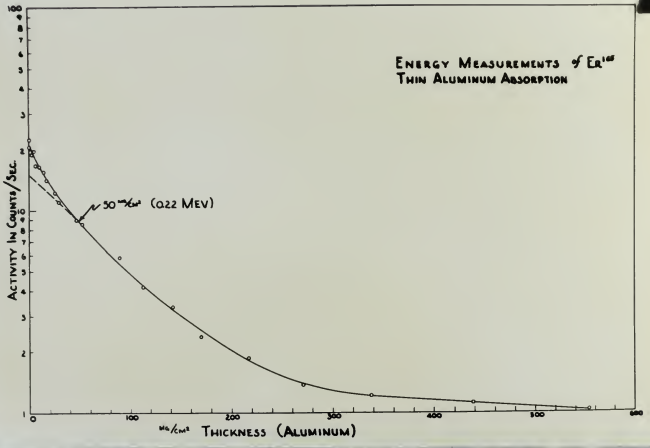
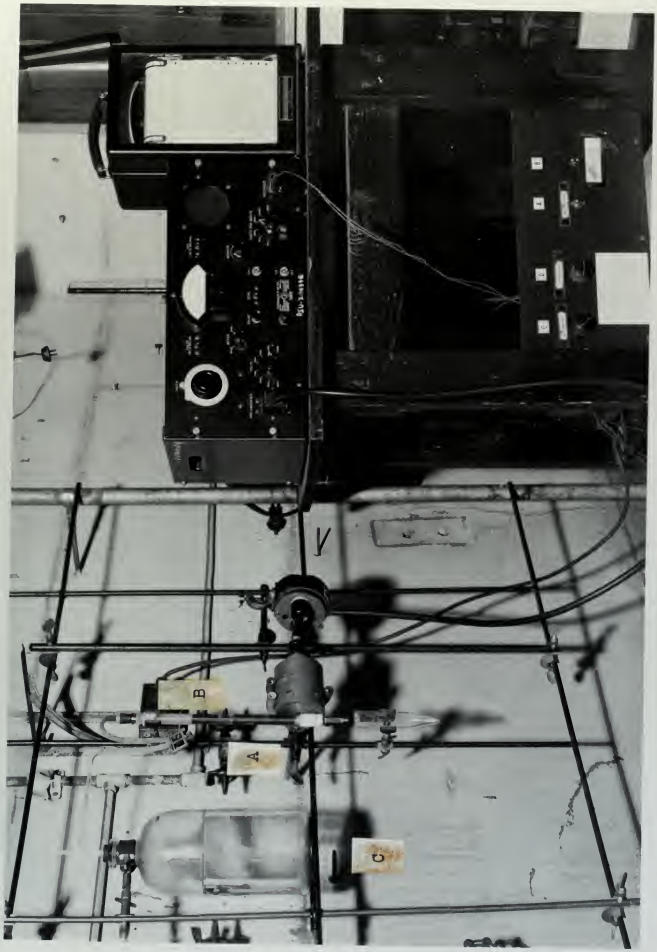
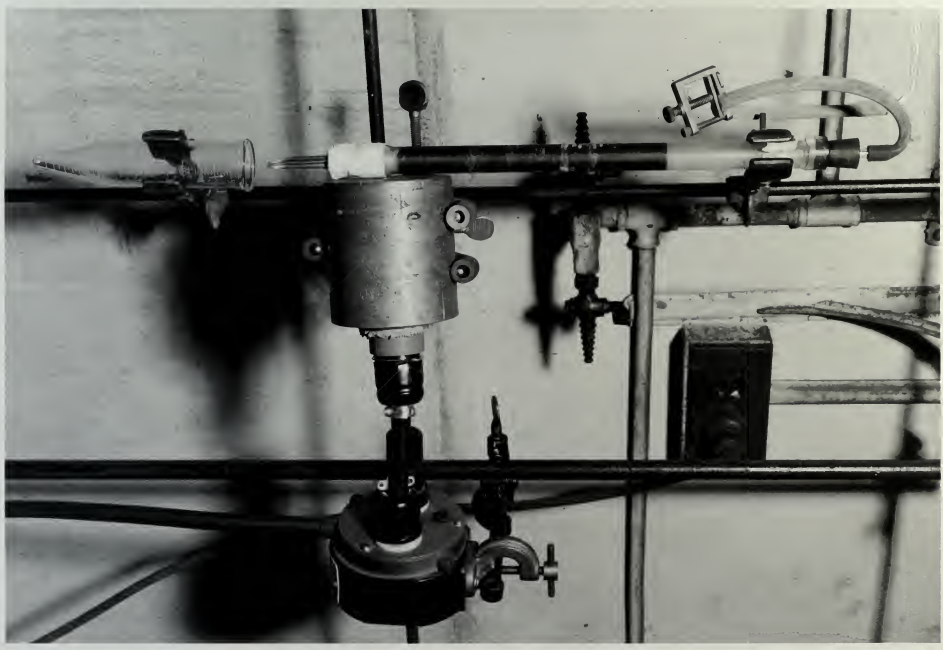

ENERGY MEASUREMENTS of Er¹⁶⁹
THIN ALUMINUM ABSORPTION
ACTIVITY IN COUNTS/SEC.
50 mg/cm² (0.22 MEV)
mg/cm² THICKNESS (ALUMINUM)
0
100
200
300
400
500
600

FIGURE 12

FIGURE 13





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